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INTER-OFFICE CORRESPONDENCE

Richmond, Virginia

To: . Dr. Jane Y. Lewis Date: January 9, 1990

From: . Robert A. Forte

Subject: . Accomplishments for 1989

## I. CROSS-TRAINING

Objective: To become familiar with the people, instrumentation, and capabilities of the Analytical Research Division (ARD).

Results: During the week of June 12, I spent time at ARD meeting with the Manager, Section Leaders, and Project Leaders. I was shown, in an overview fashion, the operation of ARD and their capabilities.

## II. METHODS DEVELOPMENT AND IMPROVEMENT

### A. Anethole Analysis

Objective: To set-up the anethole analysis of filler and after-cut solution according to the methods developed by Flavor Development.

Status: All of the equipment necessary to perform this analysis arrived. Modifications to the laboratory were completed. The circulating chiller was installed with two rotometers to maintain equal flow to both stills. Two stills were assembled and are operational. We are now capable of performing the extraction of anethole on filler and have been doing so since the beginning of December. The gas chromatograph arrived the last week in December and will be installed soon. Meanwhile, the GC work will be done using Flavor Development's GC. Jeanie Myers (laboratory technician) and I were trained by Dr. Barbu Demian, the author of this method. Ms. Myers is currently handling the anethole in filler analysis.

### B. Robotic System

Objective: To have an operating robotic system in place to perform the solvent dispensing operation in the GC Laboratory.

Status: The robotic system was installed by Hudson Robotics, Incorporated, (robot vendor and system designer) during the week of March 13 and was operational by week-end. Initially some problems surfaced but were corrected by the vendor. Since that time, the robotic system functioned satisfactorily. This robotic dispensing system is in routine operation and used daily. This

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system incorporates a comprehensive set of in-house designed safety features which allow it to dispense ethanol in a safe manner. Because of these safety features, this system carries Factory Mutual's (PM's fire insurance carrier) approval. This is the first robotic system at Philip Morris that handles a flammable liquid. After several months of operation, several enhancements were incorporated into the system software to improve the robot's performance. The robotic approach command was modified to increase the vessel pick-up accuracy. The vessel transport carriage motion was also improved by a software routine. Finally, the robotic arm was directed to a safepoint after a set of samples is completed. This safeguard prevents the robotic arm from striking any obstacles when running the next set of samples. Since this system is flexible, upgrades will be made as needed. Personnel in the GC Laboratory were trained to operate the robotic system and each technician is trained as they rotate into the GC Laboratory. To date, approximately 60% of the laboratory personnel have been trained.

C. Update Gas Chromatographs

Objective: To formulate a plan for updating the gas chromatographic instrumentation, instrument control, and intra-laboratory processing in the GC and Nicotine/Water Laboratories.

Status: The problem was evaluated and with Jeff Sampson's assistance, a plan was formulated. After evaluating GCs and intra-laboratory processing devices from several vendors, Hewlett Packard (HP) was selected to supply both components to form an integral system. The GC selected was the HP5890 Series II GC with dual autosamplers (HP7673A). The intra-laboratory processor will be an HP3365 data system. A timetable was formulated to determine when each GC will be replaced.

## III. TECHNICAL SUPPORT

A. Electrostatic Precipitation Tubes

Objective: To develop a technique to handle nicotine and water analyses using the electrostatic precipitation tubes from the DIN smoking procedure.

Status: A procedure was developed to add solvent, perform the extraction, and perform the nicotine and water analysis on smoke samples from electrostatic precipitation tubes (DIN method). The initial work was done using Monitor #25 smoke runs. The technique was refined to minimize atmospheric water contamination. A blank (unsmoked, clean tube) was analyzed daily to correct for any residual water and any atmospheric contamination due to technique. Consistency was established with these monitor runs. Several cigarette brands with different deliveries were smoked by the DIN method to establish a comparison between the nicotine and tar values generated from this method and from the FTC method.

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B. CF Analysis

Objective: To train Ron Buckner to operate the Mass Selective Detector (MSD) to perform the CF analysis (dihydro-CF, CF, 6-methyl-CF) and to quantitate the CF analogs present.

Status: Ron Buckner's training is continuing on the MSD. He has become skilled at tuning, operating, and evaluating the data acquired. He has successfully performed a calibration, interpreted, and quantitated the CF analogs. He has evaluated samples under my supervision. Ron Buckner has become quite skilled in the operation of the MSD. He has been shown the preventative maintenance procedures. He has also been taught to recognize and assess instrumental problems, as the symptoms dictate and take the necessary corrective action. Ron Buckner will continue to acquire experience with this technique and instrumentation, to learn the intricacies of the system.

C. U.S. Testing

Objective: To obtain U.S. Testing's certification on nicotine from ART smoke runs.

Status: Many ART nicotine in smoke runs were analyzed in duplicate with standard additions (spiked) on 4 and 8 mg models to comply with the requirements for U.S. Testing's certification. In addition, an extraction study was conducted on nicotine smoke extracts from a one and a four hour extraction to insure that all of the nicotine is extracted after the first hour. This work was done in duplicate with the odd ports spiked. No additional nicotine was found after the four hour extraction proving to U.S. Testing's satisfaction that a one hour extraction is sufficient. U. S. Testing has certified this method and issued a report entitled, "An Improved Technique for Accurate Measurement of Low-Level Nicotine Delivery in Cigarette Smoke," dated 9/13/89.

D. Wide Bore and Capillary Chromatography

Objective: To train the laboratory personnel in the calibration and use of wide bore and capillary chromatography.

Status: The laboratory personnel in the GC Laboratory were taught to perform the following analyses on a DB-WAX wide bore column: humectants in filler and smoke, triacetin in smoke and fumarate and succinate in cigarette papers. In the Nicotine/Water Laboratory, the personnel were taught to perform the nicotine in smoke analysis on Project ART samples using a Stabilwax-DB capillary.

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E. Mass Selective Detector (MSD)

Objective: To develop an automated analysis using the Mass Selective Detector (MSD) in Selective Ion Monitoring (SIM) mode. This would eliminate the time consuming task of manually evaluating each of the mass spectra collected.

Status: Work on this project was delayed because of the large number of samples requiring CF analysis and because of time spent training Ron Buckner to run the MSD. All of the files needed have been created along with the calibration table. The report function has been set-up and some trial runs have been made. This system needs to be optimized and some parallel testing needs to be done between the scan and the SIM mode to insure agreement.

## IV. MISCELLANEOUS

### A. Presentations and Meetings

1. Pittsburgh Conference, Atlanta, Georgia, March 6-10.
2. Presentation at CTSD Project Meeting, "FM Approved Automated Robotic Dispensing System," in-house, March 28.
3. Presentation at CTSD Project Meeting, TCRC dry run, "Laboratory Robotic Dispensing System," in-house, August 30.
4. R&D Technical Seminar, "Laboratory Robotic Dispensing System," in-house, September 20.
5. TCRC, Richmond, Virginia, October 3-4.
6. TCRC Presentation, "Laboratory Robotic Dispensing System," Richmond, VA, October 4.

### B. Completion Reports and Memos

1. Completion Report, "Laboratory Robotic Dispensing System," R. A. Forte, R. W. Kanipe and J. Y. Lewis, Acc. No. 89-042, June 6.
2. Completion Report, "Determination of Nicotine in Smoke for Alkaloid Reduced Tobacco Samples," J. Y. Lewis, R. A. Forte, J. A. Sampson, Acc. No. 89-036, July 21.
3. Memo to Mr. F. A. Lowman, "CF Analysis of Cigarettes from the Dominican Republic," February 16.
4. Memo to Mr. F. A. Lowman, "CF Analysis of Cigarettes from Mexico," February 21.
5. Memo to Mr. F. A. Lowman, "CF Analysis of Cigarettes from Brazil-August, 1988 Production," March 6.

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6. Abstract for TCRC, "Laboratory Robotic Dispensing System," March 31.

7. Memo to Mr. J. E. Wickham, "Humectant and Plasticizer Levels of Commercial Cigarettes - 1988," March 31.

8. Memo to Mr. F. A. Lowman, "CF Analysis of Cigarettes from Mexico - January, 1989 Production," April 6.

9. Memo to Ms. C. C. Bright, "CF Analysis of Cigarettes from Brazil - August, 1988 Production," May 10.

C. Training Courses

1. "Introduction to RS/1," in-house, February 7-9.

2. "Advanced Performance Appraisal," in-house, July 19.

3. J & W Scientific Technical Seminar, in-house, August 9.

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Robert A. Jones

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